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TRANSPARENT ELECTRICALLY CONDUCTIVE COATINGS

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TRANSPARENT ELECTRICALLY CONDUCTIVE COATINGS

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[There are no amendments in this patent]

Claims /1*

1. A transparent electrically conductive coating, characterized in that it comprises electrically conductive fine particles surface treated with a carbon-containing silane coupling agent and a vehicle as main components.

2. A transparent electrically conductive coating according to Claim 1, wherein the carbon-containing silane coupling agent treatment is carried out by a compound with formula (A) shown below.

$$R^1R^2_aR^3_bSiO_{(3-a-b)} \qquad (A)$$

(where R^1, R^2, and R^3 represent independently a hydrogen, alkyl, alkenyl, aryl, or hydrocarbon group having at least one substituent selected from halogen, epoxy, glycidoxy, amino, mercapto, methacryloxy and cyano group, and a and b are 0 or 1)

Detailed explanation of the invention

Industrial application field

The present invention relates to transparent coatings superior in surface hardness and having electrical conductivity and antistatic property. Especially, it relates to transparent electrically conductive coatings superior in durability because the coatings can be made thick.

Prior art

As transparent electrically conductive coatings, metallic thin film such as gold, silver, aluminum, and the like, tin oxide, antimony-doped tin oxide, or composite oxide (abbreviated as ITO) of indium oxide and tin oxide is most well known. On the other hand, in antistatic coatings, various ionic compounds such as cationic or anionic compounds beside the electrically conductive coatings have been known.

Problems to be solved by the invention

Transparent electrically conductive coatings comprising metal films such as gold, silver, aluminum, and the like have such drawbacks as low hardness and absorption of visible rays. Further, in coatings comprising tin oxide, ITO etc., the productivity is extremely bad in thick layer formation since they are formed generally by a method such as sputtering, thermal CVD, vacuum deposition, and the like, and thus they are not practical from the aspect of cost. Namely, the transparent electrically conductive films have a big drawback of being easily scratched or /2

* [Numbers in the right margin indicate pagination in the original foreign language text.]

breaking when the films collide with a slightly hard material or are worn away since the transparent electrically conductive films are extremely thin films comprising metal thin films or metal oxide thin films.

On the other hand, the antistatic coatings utilizing ionic compounds have such a drawback that their antistatic efficiency is greatly controlled by environmental changes such as temperature, etc., and especially at low temperature, their function is not displayed.

The purpose of the present invention is to provide transparent electrically conductive coatings having high surface hardness, excellent durability and no efficiency deterioration even at low temperature.

Means to solve the problem

The present invention for accomplishing the aforementioned purpose has the following constitution.

"Transparent electrically conductive coatings, characterized in that the coatings comprise electrically conductive particles surface treated with a carbon-containing silane coupling agent and vehicles as main components."

In the present invention, there is no restriction on base materials, on which the transparent electrically conductive coatings are formed, if these require the present invention, but it is preferred to apply the coatings to base materials having transparency from the standpoint that the coatings obtained by the present invention have transparency. Further, as the base materials, inorganic glass or organic plastics may be used without any problem. Furthermore, the shape of the base materials is not restricted to any specific one and it may be a flat sheet, a curved one or circular one. Further, even those obtained by pre-forming a transparent electrically conductive film comprising a thin metal film or metal oxide film such as ITO, tin oxide, and the like on the whole surface or a portion of base materials are preferred for the devices for use in handwriting input devices and the like.

In the present invention, the electrically conductive particles are not particularly restricted if they provide sufficient transparency and electrical conductivity to the coatings, but tin oxide (including antimony-doped tin oxide), indium oxide/tin oxide mixture, and the like are exemplified as preferable examples from the standpoint that they easily satisfy both the high electrical conductivity and transparency. Furthermore, the average particle diameter of the electrically conductive fine particles is not restricted if the aforementioned purpose is accomplished, but those having a primary particle diameter of 10Å to 5 μm are preferred from the standpoint that the disperse state is stabilized and a homogeneous coating is easily obtained. Here, the primary particle diameter means a particle diameter able to be dispersed by any dispersing method. Further, it is preferred that the electrically conductive fine particles be

included generally at an amount of 30-90 wt% in the coatings, although it may vary with the conductivity and transparency of the desired coatings. Namely if it is less than 30 wt% sufficient conductivity cannot be obtained, further if it exceeds 90 wt%, the appearance such as transparency and adhesiveness to base materials tend to be deteriorated.

It is necessary for the electrically conductive fine particles to be used in the present invention to treat the particle surface with a carbon-containing silane coupling agent. Namely, it is due to that the dispersibility of the electrically conductive fine particles becomes satisfactory and their surface hardness is improved by treatment with the silane coupling agent. As the state of treated fine particles, the fine particles covered with silane coupling agent in the chemically bonded state is preferred from the standpoint that the treatment stability and treatment effect are high. Further, they may be coated with a coating obtained by a silane coupling agent.

The carbon-containing silane coupling agent has at least a carbon-containing organic group by –Si-C- bond, and the treatment amount of the carbon-containing silane coupling agent for the electrically conductive fine particles should be decided by the relation between particle diameter and vehicle, but it is preferred to use at an amount of 0.01 part by weight or more, more preferably 0.05 part by weight or more, per 100 parts by weight of the electrically conductive fine particles in order to reveal the treatment effect, especially the surface hardness improving effect of the coatings. Further, it is preferred to use it at an amount of 5.00 parts by weight or less, more preferably 3 parts by weight or less, per 100 parts by weight of the electrically conductive fine particles from the viewpoint of conductivity retention.

As the electrically conductive fine particles treated with a carbon-containing silane coupling agent, it is preferred that the silane coupling agent exist on the surface of the treated electrically conductive fine particles as the functional group expressed by the formula (A) shown below. /3

$$R^1R^2_aR^3_bSiO_{(3-a-b)} \qquad \text{(A)}$$

(Where R^1, R^2, and R^3 represent independently a hydrogen, alkyl, alkenyl, aryl, or hydrocarbon group having at least one substituent selected from halogen, epoxy, glycidoxy, amino, mercapto, methacryloxy and cyano group, and a and b are 0 or 1.)

Various methods can be applied for treatment by the silane coupling agent. Especially, it is most preferable to treat an organosilicon compound with formula (A) capable of producing functional groups, for example, an alkoxysilane having a substituent suited to the formula (A), particularly a lower alkoxysilane such as methoxysilane, ethoxysilane, and the like, acyloxysilane such as acetoxysilane, halogenosilane such as chlorosilane, organosilicon compound having hydrolyzable functional groups such as a silazane compound, ketoximesilane, isopropenylsilane, and the like, in a gas phase, solvent-free state or liquid phase in various

solvents and to initiate the hydrolysis during or after the treatment to carry out the surface treatment due to that it is simple, and uniform treatment is possible. Furthermore, the treatment at high temperature is effective.

The present invention is a process of including electrically conductive fine particles surface treated with the aforementioned carbon-containing coupling agent in various vehicles and coating, and any vehicle can be used here if the electrically conductive fine particles can be dispersed in the vehicle. However, it is preferred to use organosilicon compounds with the formula (B) shown below and/or their hydrolyzates as vehicles having good affinity with the electrically conductive fine particles and providing high hardness due to that the electrically conductive fine particles are silane coupling agent-treated inorganic substances, especially inorganic oxides.

$$R^4_cR^5_dSi(X)_{4-c-d} \qquad (B)$$

(Where R^4 and R^5 represent independently an alkyl, alkenyl, aryl, or hydrocarbon group having a halogen, epoxy, glycidoxy, amino, mercapto, methacryloxy and cyano group; X represents a hydrolyzable group; and c and d are 0 or 1.)

Typical examples of organic silicon compounds expressed by the formula (B) and/or their hydrolyzates include tetraalkoxysilanes and their hydrolyzates such as methyl silicate, ethyl silicate, n-propyl silicate, isopropyl silicate, n-butyl silicate, sec-butyl silicate, tert-butyl silicate, and the like; trialkoxysilanes, triacyloxysilanes or triphenoxysilanes such as methyltrimethoxysilane, methyltriethoxysilane, methyltrimethoxysilane, methyltriacetoxysilane, methyltributoxysilane, ethyltrimethoxysilane, ethyltriethoxysilane, vinyltrimethoxysilane, vinyltriethoxysilane, vinyltriacetoxysilane, vinyltrimethoxyethoxysilane, phenyltrimethoxysilane, phenyltriethoxysilane, phenyltriacetoxysilane, γ-chloropropyltrimethoxysilane, γ-chlorotriethoxysilane, γ-chloropropylacetoxysilane, 3,3,3-trifluoropropyltrimethoxysilane, γ-methacryloxypropyltrimethoxysilane, γ-aminopropyltrimethoxysilane, γ-aminopropyltriethoxysilane, γ-mercaptopropyltrimethoxysilane, γ-mercaptopropyltriethoxysilane, N-β-(aminoethyl)-γ-aminopropyltrimethoxysilane, β-cyanoethyltriethoxysilane, methyltriphenoxysilane, chloromethyltrimethoxysilane, chloromethyltriethoxysilane, glycidoxymethyltrimethoxysilane, glycidoxymethyltriethoxysilane, α-glycidoxyethyltrimethoxysilane, α-glycidoxyethyltriethoxysilane, β-glycidoxyethyltrimethoxysilane, β-glycidoxyethyltriethoxysilane, α-glycidoxypropyltrimethoxysilane, α-glycidoxypropyltriethoxysilane,β-glycidoxypropyltrimethoxysilane, β-glycidoxypropyltriethoxysilane, γ-glycidoxypropyltrimethoxysilane, /4

γ-glycidoxypropyltriethoxysilane, γ-glycidoxypropyltripropoxysilane, γ-glycidoxypropyltributoxysilane, γ-glycidoxypropyltrimethoxyethoxysilane, γ-glycidoxypropyltriphenoxysilane, α-glycidoxybutyltrimethoxysilane, α-glycidoxybutyltriethoxysilane, β-glycidoxybutyltrimethoxysilane, β-glycidoxybutyltriethoxysilane, γ-glycidoxybutyltrimethoxysilane, γ-glycidoxybutyltrimethoxysilane, δ-glycidoxybutyltrimethoxysilane, δ-glycidoxybutyltriethoxysilane, (3,4-epoxycyclohexyl)methyltrimethoxysilane, (3,4-epoxycyclohexyl)methyltriethoxysilane, β-(3,4-epoxycyclohexyl)methyltrimethoxysilane, β-(3,4-epoxycyclohexyl)methyltriethoxysilane, β-(3,4-epoxycyclohexyl)methyltripropoxysilane, β-(3,4-epoxycyclohexyl)methyltributoxysilane, β-(3,4-epoxycyclohexyl)methyltrimethoxyethoxysilane, β-(3,4-epoxycyclohexyl)methyltriphenoxysilane, γ-(3,4-epoxycyclohexyl)propyltrimethoxysilane, γ-(3,4-epoxycyclohexyl)propyltriethoxysilane, δ-(3,4-epoxycyclohexyl)butyltrimethoxysilane, δ-(3,4-epoxycyclohexyl)butyltriethoxysilane, and the like; and dialkoxysilanes or diacyloxysilanes or their hydrolyzates such as dimethyldimethoxysilane, phenylmethyldimethoxysilane, dimethyldiethoxysilane, phenylmethylethoxysilane, γ-chloropropylmethyldimethoxysilane, γ-chloropropylmethyldiethoxysilane, γ-methacryloxypropylmethyldimethoxysilane, γ-methacryloxypropylmethyldiethoxysilane, γ-mercaptopropylmethyldimethoxysilane, γ-mercaptopropylmethyldiethoxysilane, γ-aminopropylmethyldimethoxysilane, γ-aminopropylmethyldiethoxysilane, methylvinyldimethoxysilane, methylvinyldiethoxysilane, glycidoxymethylmethyldimethoxysilane, glycidoxymethylmethyldiethoxysilane, α-glycidoxyethylmethyldimethoxysilane, α- glycidoxyethylmethyldiethoxysilane, β-glycidoxyethylmethyldimethoxysilane, β- glycidoxyethylmethyldiethoxysilane, α-glycidoxypropylmethyldimethoxysilane, α-glycidoxypropylmethyldiethoxysilane, β-glycidoxypropylmethyldimethoxysilane, β-glycidoxypropylmethyldiethoxysilane, γ-glycidoxypropylmethyldimethoxysilane, γ-glycidoxypropylmethyldiethoxysilane, γ-glycidoxypropylmethyldipropoxysilane, γ-glycidoxypropylmethyldibutoxysilane, γ-glycidoxypropylmethyldimethoxyethoxysilane, γ-glycidoxypropylmethyldiphenoxysilane, γ-glycidoxypropylethyldimethoxysilane, γ-glycidoxypropylethyldiethoxysilane, γ-glycidoxypropylethyldipropoxysilane, γ-glycidoxypropylvinyldimethoxysilane, γ-glycidoxypropylvinyldiethoxysilane, γ-glycidoxypropylphenyldimethoxysilane, γ-glycidoxypropylvinyldiethoxysilane, and the like.

These organosilicon compounds can be used singly or in combination of two or more.

It is preferred to use these organosilicon compounds by hydrolyzing in order to cure at a lower curing temperature.

Hydrolysis is carried out by adding water or an acidic aqueous solution such as hydrochloric acid, acetic acid or sulfuric acid to the organosilicon compounds and stirring. Furthermore, the degree of hydrolysis can be easily controlled by adjusting the addition amount of pure water or acidic aqueous solution. It is especially preferred in the hydrolysis to add pure water or acidic aqueous solution at an equimolar amount or more but a 3 times molar amount or less of the hydrolyzing group of formula (B) is preferred from the standpoint of curing acceleration.

Since alcohol, etc., is produced in hydrolysis, it is possible to hydrolyze the organosilicon compound in solvent-free state, but it is also possible to hydrolyze it after mixing with a solvent for homogeneous hydrolysis. Further, it is also possible to remove a proper amount of alcohol after hydrolysis by heating and/or treating in a vacuum according to the purpose. As these solvents, alcohols, esters, ethers, ketones, halogenated hydrocarbons or aromatic hydrocarbons such as toluene, xylene, and the like are exemplified. Further, these solvents may be used as mixed solvents of two or more if necessary. Further, it is possible to heat it above room temperature in order to accelerate hydrolysis reaction and carry out the reaction such as precondensation, etc., according to the purpose and, needless to say, it is possible to carry out the hydrolysis reaction below room temperature in order to prevent precondensation.

The curing of the organosilicon compounds and/or their hydrolyzates is carried out generally by heating, but various curing catalysts are added for shortening of heating time and low-temperature curing. As the curing catalysts, alkali metal salts of carboxylic acids, alkoxides of titanium, zirconium, and the like or chelate compounds are exemplified.

Particularly, aluminum chelate compounds represented by acetylacetone aluminum salt are preferably used due to the stability and transparency of the compositions and no coloration.

Further, in the organosilicon compounds with the formula (B), those having, as R^4 and R^5, a C_{1-4} alkyl, alkenyl or phenyl, or organic group having an epoxy or glycidoxy group are preferred from the standpoint of adhesion with base materials, surface hardness improvement, heat resistance, weathering resistance, and the like.

Dielectric colloidal inorganic oxides are further added to the aforementioned vehicles from the standpoint of hardness improvement and improvement of adhesion to inorganic base materials, especially inorganic glass or inorganic glass having an ITO coating or tin oxide coating. As concrete examples of the dielectric colloidal inorganic oxides, metal oxides from Si, Ti, Al, Zr, Sb, W, Ta and the like are exemplified, especially those having an average particle diameter of 1 nm to 200 nm are preferably used. Of these, the Group Va metal oxides such as Sb are preferable for improvement of electric conductivity.

As means for the formation of coating on transparent base materials of the present invention, an ordinary coating method such as brush coating, dip coating, roll coating, screen printing process, spray coating, spin coating, flush coating, and the like can be easily applied.

The thickness of coatings is not particularly restricted in the present invention, but a thickness of 0.1-50 μm is preferably used from the standpoint of retaining of adhesion strength, hardness and the like. Particularly preferred thickness is 0.5-20 μm. Further, in the formation of coating, the coating materials are diluted with various solvents and used for workability, control of coating thickness, and the like, and as the dilution solvent, water, alcohols, esters, ethers, halogenated hydrocarbons, dimethyl formamide, dimethyl sulfoxide, and the like can be used according to the purpose, and a mixed solvent can be used if necessary. Further, when colloidal inorganic oxides such as antimony oxide and the like are used, water, alcohols, dimethyl formamide, ethylene glycol, diethylene glycol, triethylene glycol, benzyl alcohol, phenethyl alcohol, phenyl Cellosolve, and the like are preferably used from the standpoint of dispersibility.

Beside the aforementioned silicon compounds as the vehicle component in the present invention coatings, various epoxy resins, melamine resin, acrylic resins, urethane resin, polyvinyl acetate and its saponified product, nylon resin, and polyester resin can be used within such a range of not largely damaging the transparency and not lowering the hardness. Further, the /6
content of the vehicle in the coating is preferably 10-70 wt%. If it is less than the above range, a problem in appearance, adhesion to base materials, and the like occurs, and if it exceeds the above range, there is a problem of deterioration of the electrical conductivity.

The coating of the present invention is generally formed by coating compositions comprising the aforementioned various components on base materials.

In the formation of coatings from these compositions, generally, coating compositions containing the above compositions are coated, dried by heating and cured.

The curing temperature is 60-300°C and preferably 80-200°C, although it is varied with the composition to be selected, working condition, base materials, and the like. If the curing temperature is lower than the above range. insufficient curing results, and if it is higher than said range, problems such as crack formation, decomposition of the coating, and the like occur.

The electrical conductivity and transparency of coatings obtained by the present invention should be optimized according their application purposes, but when application to conventional transparent base materials and liquid-crystal input devices is considered, a specific resistivity of 5×10^{5} Ω·cm or lower and a haze value of 30% or less are preferably used. On the other hand, in the case of an antistatic purpose, a specific resistivity of 5×10^{6} Ω·cm or lower, more preferably 1×10^{6} Ω·cm or lower, and a haze value of 80% or less, more preferably 40% or less, are preferably used. Further, in the case where an electrically conductive pen such as a pencil is used in the application to an input device, a surface roughness of the coating of less than

2 μm is preferred from the sense of leaving no drawing mark or of easy removal of the drawing mark. The surface roughness is measured by the method prescribed in JIS B0651.

As the dispersing method of each component in the present invention, even stirring by stirring blades is sufficient, but a paint conditioner, sand mill, three-roll mill, ball mill, homomixer, homogenizer, and the like are preferably used for making the dispersed state satisfactory.

On the other hand, various surfactants are added or applied for keeping the dispersed state stable, especially nonionic surfactants such as silicone surfactants, fluorine type surfactants or phenyl ether surfactants, and the like are preferably used.

Further, transparent electrically conductive coatings not reflecting color tone can be obtained by adding dyes that can reinforce the color tone of coatings.

The transparent electrically conductive coatings obtained by the present invention are superior in transparency and surface hardness and can be used for window glass, the front plates of various devices such as CRT or liquid crystals, or input device parts for the above-mentioned devices.

Application examples

Next, application examples are exemplified in order to make the purposes of the present invention clear, but the present invention is not limited to these application examples only.

Application Example 1

(1) Preparation of silane hydrolyzate

A silane hydrolyzate was obtained by cooling 30.2 g γ-glycidoxypropyltrimethoxysilane to 10°C, slowly dropping 6.9 g 0.01N aqueous hydrochloric acid solution under stirring, and continuously stirring at room temperature for 1 h.

(2) Preparation of coating agent

The aforementioned silane hydrolyzate was mixed with 7.1 g bisphenol A type epoxy resin (Epikote 827, Shell Chemical Co.), 22.1 g N,N-dimethyl formamide, 10.0 g benzyl alcohol, 17.1 g methyl alcohol, and 1.5 g silicone surfactant, furthermore 29.7 g colloidal antimony pentoxide sol (Antimony sol A-2550, Nissan Chemical Co.) and 2.2 g acetylacetone aluminum salt were added and sufficiently stirred.

Furthermore, 10.0 g indium oxide/tin oxide mixed inorganic oxide (ITO fine particles) having average particle diameter of 0.1 μm, the surface of which was pretreated with trimethylmethoxysilane (carbon content 0.5%), as electrically conductive fine particles were

added, followed by adding 395 g phenethyl alcohol and sufficiently dispersed in homogenizer to obtain a coating agent. /7

(3) Coating and curing of the coating agent

The above coating agent was coated on a 3.0-mm-thick flat glass substrate using #16 bar coater, set in a room for 5 min, and thermally cured at 80°C for 12 min and at 130°C for 2 h.

(4) Evaluation method

(a) Coating layer thickness

It was evaluated in accordance with JIS B0651.

(b) Surface hardness

The pencil hardness was measured in accordance with JIS K5400 and used as the surface hardness.

(c) Permeability, haze

It was measured using a haze computer (Suga Shikenki Co., HGM-2DP).

(d) Sheet surface resistivity

The surface resistivity was determined by the four-probe method.

(5) Test result

The transparent electrically conductive coating thus obtained had satisfactory transparency, slight coloration and preferable appearance. Further, it was confirmed that it had extremely satisfactory antistatic effect by carrying out an abrasion electrification test with cloth at low humidity of 20% relative humidity. Other physical properties are shown in Table 1.

<u>Application Example 2</u>

An electrically conductive coating was obtained in the same manner as in Application Example 1 except that a 1-mm-thick transparent conductive film-coated glass (Nesa glass 110 Ω/□) was used as the substrate and the spin coater process, instead of bar coater process, was used. The electrically conductive coating had excellent transparency as in Application Example 1, and a continuity stable throughout the entire coating surface against the drawing input from the coating surface was obtained between electrically conductive films. Further, when drawing using a pencil with hardness B was carried out on it, no scratch was formed even after 10,000 times of drawing with the pencil, and a protective layer having excellent drawing durability was obtained. Other physical properties are shown in Table 1.

Application Example 3

An electrically conductive coating was obtained in the same manner as in Application Example 2 except that the surface of electrically conductive particles was treated with hexamethyldisilazane (carbon content 1.0%). The transparent electrically conductive coating was a protective layer superior in drawing durability as in Application Example 2.

Application Example 4

A coating agent was obtained in the same manner as in Application Example 1 except that 0.012 g PTB-31 (CI number Disperse-26) and 0.063 g PTV-57 (CI number Disperse-31) as pigments for shading for compensating the color tones of the electrically conductive fine particles and vehicle were additionally added to slightly reduce coloration from the electrically conductive fine particles and vehicle and sufficiently dispersed in a homogenizer. The coating was colorless and transparent, and the coloration degree (YI value) was decreased to 1/2. Further, it was confirmed that the coating was a satisfactory antistatic coating as in Application Example 1. Other physical properties are shown in Table 1.

Comparative Example 1

A transparent electrically conductive coating was obtained in the same manner as in Application Example 1 except that the indium oxide/tin oxide mixed inorganic oxide (ITO fine particles), which was not surface treated, was used. The transparent electrically conductive coating had a surface hardness of 6H, which was slightly deficient. Other physical properties are shown in Table 1.

Comparative Example 2

A transparent coating was obtained in the same manner as in Application Example 2 except that ITO particles were used without surface treatment. The coating was superior in transparency and also continuity property, but the surface hardness was slightly deficient as 5H.

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Table 1

Example No.	Appearance	Evaluation results				
		Thickness (μm)	Permeability (%)	Haze (%)	Sheet resistance (Ω/□)	Pencil hardness
Application Example 1	Slightly yellow, transparent	2.5	84.0	2.0	$5x10^5$	9H
Application Example 2	Slightly yellow, transparent	1.1	84.5	1.8	$2x10^2$	9H
Application Example 3	Slightly yellow, transparent	1.1	84.8	1.7	$2x10^2$	9H
Application Example 4	Colorless, transparent	2.5	83.2	2.0	$8x10^5$	9H
Comparative Example 1	Slightly yellow, transparent	2.5	83.8	2.2	$3x10^5$	6H
Comparative Example 1	Slightly yellow, transparent	1.1	84.8	1.8	$2x10^2$	5H

Effect of the invention

The transparent electrically conductive coatings obtained by the present invention have the following effects.

(1) Coatings having high surface hardness are formed, and the drawing durability is superior.

(2) Coatings are useful for input devices due to high transparency and excellent electrical conductivity.

(3) The antistatic property is excellent event at low humidity.